

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant	: Borzatta et al.
App. No	: 10/577,409
Filed	: October 26, 2004
For	: FORMULATION OF A SYNERGISTIC INSECTICAL COMPOSITION
Examiner	: Pack, John.
Art Unit	: 1616
Conf No.	: 1407

DECLARATION UNDER 37 C.F.R §1.132

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

1. I, Valerio Borzatta, am an inventor of the present application and I have extensive experience in the field of pesticides for many years.

2. I duly sworn depose and say that:

- I am an Italian citizen residing at: Bologna (Italy) Via Bellettini 20 - 40127
- I am familiar with the English language.
- I graduated in: CHEMISTRY in the academic year: 1970
- I am co-inventor of following US patents/applications: 7,402,709; 11/912,136; 11/912,299; 11/629,182; 7,544,843; 10/577,409; 10/504,367; 7,019,154; 6,252,092; 6,342,613;
- My Previous job experiences are:
 - (i) Head of no antibiotic products lab in Alfa Wasserman

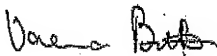
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(ii) Head of chemical research and patents in Ciba Geigy, Italy

- My actual job is R&D Director at Endura S.p.A

3. The experimental tests shown in Annex A, appended herewith have been carried out under my own responsibility. The tests concerns the results of preparation of Formulation of Example 7 of US3,846,551 in comparison with formation of complex according to US2007/0072827.

4. I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or patent issuing therefrom.



Dated: February 6, 2012

By: _____

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Experimental part

First of all, the sample of bifenthrin in β -CD was prepared by kneading, following the general procedure reported in US 3,846,551 of Mifune et al in order to prepare the interacted compound (column 5, lines 27-37). The interacted compound of bifenthrin in β -cyclodextrin was added with PBO as in Example 7 of the above mentioned Patent, being the only difference the use of bifenthrin instead of furamethrin.

A further sample of the complex bifenthrin, PBO in β -CD was prepared as in Example 6 of US 2007/0072827 (Patent application n. 10/577,409)

Samples of 1 mg/ml were prepared in D₂O solution and all ¹H NMR spectra were recorded using a 5 mm tube in D₂O without degassing , 1 ml of suspension in each tube. ¹H NMR spectra of β -cyclodextrin (β -CD) complexes were recorded with a Bruker Avance 400 spectrometer at 400-13 MHz, temperature 298°K.

NMR parameters were :

Time domaine size : 64K

Spectral width : 4,000HZ

Pulse width: 8.2 μ s

Acquisition time : 8.18 sec

Relaxation delay : 1s

Number scans : 32

Chemical shifts are given in ppm (δ) which were measured relative to the peak of the solvent D₂O .

¹H-NMR spectrometry is a good diagnostic tool as well as useful in the study and characterization of β -CD inclusion complexes, specifically in order to prove the formation of the β -CD insecticide inclusion complex .

The results of the two prepared samples are reported in Table 1 and in Table 2.

In particular, Table 1 shows the chemical shifts of H3 and H5 β -CD protons, located into lipophilic cavity as signals of the formation of inclusion complexes.

As in Table 1, we can observe that the preparation of interacted complex of bifenthrin in β -CD and subsequent kneading of the preformed complex with PBO as in Example 7 of US 3,846,551 gives four different chemical peaks, two of them characteristic of a complex formation (3.710 ppm and 3.650 ppm) whilst the other two peaks being characteristic of β -CD proton without any complexation.

This means a partial complexation of bifenthrin by using kneading methods with beta-CD free and bifenthrin free in the final sample.

On the contrary, following the procedure for the joint complexation of bifenthrin and PBO according to Example 6 of US 2007/0072827 (Patent application n. 10/577,409), results show that a complex was formed with no bifenthrin and beta-CD free in the mixture.

In Table 2 the chemical shifts of O-CH₂-O protons of PBO are shown. The chemical shift of 5.465 ppm is characteristic of free (no complexed) PBO.

As clearly shown in Table 2, following the kneading procedure of Mifune, PBO is not complexed, while the complexation of PBO is present when the joint complex is formed in accordance with US 2007/0072827 (Patent application n. 10/577,409).

In summary, following the procedure indicated in Mifune et al, i.e. by kneading an interacted compound of pyrethroid in beta-CD with PBO, there is no joint complexation of PBO in beta-CD, contrarily to the procedure stated in US2007/0072827, according to which all the PBO present is complexed in beta-CD jointly to the pyrethroid.

Table n. 1

	β CD Proton H3 (ppm)	β CD Proton H3 (ppm)	β CD Proton H5 (ppm)	β CD Proton H5 (ppm)	H3 $\Delta\delta$ /H5 $\Delta\delta$ β CD free- β CD complex*
β CD free		3.822		3.715	-----
β CD bifenthrin complex + PBO (after kneading) as in US 3,846, 551	3.710	3.822	3.650	3.715	-0.120/0/0.065/0
β CD bifenthrin + PBO complex as in US Patent Application 10/577,409	3.701	-----	3.600	-----	-0.120/-0.115

* - $\Delta\delta$: lower field; + $\Delta\delta$: higher field

Table n.2

	$\text{OCH}_2\text{O}_{\text{free}}$ (proton, multiplicity)	$\text{OCH}_2\text{O}_{\text{no free}}$ ppm
PBO Free	5.465 (2H, s)	-----
β CD bifenthrin complex + PBO (after kneading) as in US 3,846, 551	5.469 (2H, s)	-----
β CD bifenthrin + PBO complex as in US Patent Application 10/577,409	5.360 (2H, s)	5.660 (2H, s)